

# CHEMICAL METALLURGY

Ref: University of Pittsburgh, College in High School, Lab Manual, 1998

## INTRODUCTION

Most reactions occur in solution and many occur extremely quickly. The reactions in this lab are in their solid states. These are examples of metallurgy, because the products include elemental metals. They are also single replacement reactions and redox reactions.

We shall see two common features of reactions of solids. First, they are often slow at room temperature and so heating is required. In the thermite reaction, the initial heating is provided by burning magnesium “fuse”. Secondly, the chemical conversion may be less than 100% efficient. Not all the metal oxide may react in the desired fashion and, even if it does, it may be difficult to recover the entire elementary metal product. What product yield(recovery rate) can you achieve: 20%, 60%, 95%? This is a practical concern in the chemical industry, because a low recovery rate means waste and gives rise to pollution due to the unrecovered material.

In order to maximize the product yield of lead and iron, the reagents, carbon and aluminum must be in excess. The amounts given in the procedural instructions ensure this.

## SAFETY

Crucibles heated to red heat take at least 15 minutes to cool. They can cause serious burns even when they don't look hot! Be very careful what you pick up and how – use tongs to be safe.

One reaction involves ignition of a highly exothermic mixture. The actual ignition should be done by the instructor, who will first confirm the conditions are safe.

This lab does involve lots of heat and sparks which could cause fires. Have a Fire Extinguisher and someone who knows how to use it standing-by.

Disposal: Place solid waste in “solid waste” can.

Wear your apron and goggles during this experiment.

## MATERIALS

Lead (II) Carbonate 6g  
12.5 cm Filter Paper  
Iron (II) Oxide 20g  
400 ml Beaker  
Aluminum 8.0g  
Crucible tongs  
Pan (23 cm pie pan)  
Ring stand

Sand  
Porcelain Crucible  
Clay triangle  
Iron ring  
Fume Hood  
Fisher burner  
50 ml beaker

## PROCEDURE

### Part A. Preparation of lead by reaction of lead (II) oxide with carbon.

You will use lead (II) carbonate as the source of lead (II) oxide and filter paper as the source of the carbon. As the carbon is in excess, the amount is not critical (as long as it is in excess). The amount of lead (II) carbonate used is also not critical, but it must be measured to sufficient precision, in this case to a mass of 0.01 g.

Place some sand in a crucible to cover the bottom (this helps to prevent the lead from fusing with the porcelain). Weigh out about 3 g of lead (II) carbonate on two pieces of 12.5 cm diameter filter paper. Wrap the filter paper around the solid (dampening the paper is necessary) so that it will fit into the crucible with its lid in place. Place the crucible on a clay triangle supported on an iron ring under the fume hood. Position a Fisher burner underneath, with a space of about one inch between the top of the burner and the iron ring. Heat the crucible gently for a few minutes, then with maximum heat for about 30 minutes: the crucible may glow red at this stage.

Lead melts at 330°C and so your crucible should contain the liquid metal at the end of the heating period. Proceed as follows. Half fill a 400 mL beaker with water. Using crucible tongs, shake or swirl the crucible to allow the liquid droplets to coalesce. (It may be easier to grasp one of the metal wires on the clay triangle with tongs and shake it carefully). Then remove the Fisher burner, remove the crucible lid and pour the liquid contents into the beaker of water. Replace the crucible onto the clay triangle and leave it for 15 minutes to cool before handling it. When the lead is cool, dry it and weight it. Show your sample to your instructor before disposing of it.

### Part B. Preparation of iron by reaction of iron (III) oxide with aluminum (Thermite reaction)

This reaction is initiated by the high temperature produced by a burning Mg fuse. It then occurs in a few seconds. So it is essential that the solid reagents are mixed extremely well: any iron oxide that is not close to aluminum powder will not react.

Weigh out about 10 g of iron (III) oxide and about 4.0 g of aluminum, both measured to 0.01 g. The iron (III) oxide is provided in a glass vial. Weigh the vial, empty the solid into a 50 mL beaker and reweigh the vial. Mix the solids thoroughly in the beaker until the mixture is a uniform reddish-brown color. Fold two pieces of filter paper into a cone and support it in a clay triangle, placed on an iron tripod. Place the whole assembly under the hood over a pan about three-quarter full of sand. Transfer the reaction mixture to the paper cone. Twist together 3 pieces of Mg ribbon (one 5" long and two 3" long) and insert this into the mixture. Dampen the outside of the cone slightly with water.

Your instructor will light the fuse. Everyone else should keep well away. **ARE YOUR GOGGLES ON??** Have a fire extinguisher ready, in case of emergency. Leave the reaction products for 5 minutes to cool. For a final cooling, pick up the solid produce with crucible tongs and quench it in water in a 400 mL beaker. Then break up the mass and use a magnet to help to separate the iron pieces from  $\text{Al}_2\text{O}_3$  and sand. Clean the product as well as possible, then weigh it. Show your sample to the instructor before disposing of it in the solid waste container.

## DATA ANALYSIS

### Part A.

1. Find a definition and other relevant information for metallurgy, thermite process and percent yield.
2. Write a balanced equation for the decomposition of lead (II) carbonate to yield lead (II) oxide and carbon dioxide gas.
3. Write a balanced equation for the reaction of lead (II) oxide with carbon to give elemental lead and carbon dioxide.

### Measurements

3. Mass of lead (II) carbonate \_\_\_\_\_
4. Mass of elemental lead formed \_\_\_\_\_

### Literature

5. Molar mass of lead \_\_\_\_\_
6. Molar mass of lead (II) carbonate \_\_\_\_\_

### Calculations (\*-show work)

7. \*#mol of lead (II) carbonate \_\_\_\_\_
8. \* Expected # mol of lead (theoretical yield) \_\_\_\_\_
9. \*Measured # mol of lead \_\_\_\_\_
10. Percent yield of lead \_\_\_\_\_

Discussion

11. Treating the calculated percent yield of lead as an **observation**, state a valid **explanation**, **conclusion** or **question** based on the value obtained.
  
12. Describe the appearance of the lead. What metallic properties does it show?

**Part B.**

1. Write a balanced equation for the reaction of iron (III) oxide with aluminum.
  
2. Write a balanced equation showing what happens to the magnesium ribbon.

Measurements

3. Mass of vial + iron (III) oxide \_\_\_\_\_
4. Mass of vial empty \_\_\_\_\_
5. Mass of iron (III) oxide \_\_\_\_\_
6. Mass of aluminum \_\_\_\_\_
7. Mass of elemental iron formed \_\_\_\_\_

Literature

8. Molar mass of iron \_\_\_\_\_
9. Molar mass of iron (III) oxide \_\_\_\_\_

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Calculations (\*-show work)

10. \*#mol of iron (III) oxide \_\_\_\_\_
11. \*Expected # mol of iron (theoretical yield) \_\_\_\_\_
12. \*Measured # mol of iron \_\_\_\_\_
13. Percent yield of iron \_\_\_\_\_

Discussion

14. State a valid **explanation**, **conclusion** or **question** based on the observed percent yield of iron.
15. Is the mass of aluminum (#6) important? Explain carefully.
16. Describe the appearance of the iron. Does it show the properties of a metal?
17. Describe the appearance of the aluminum oxide product. Does it show the properties of a metal?